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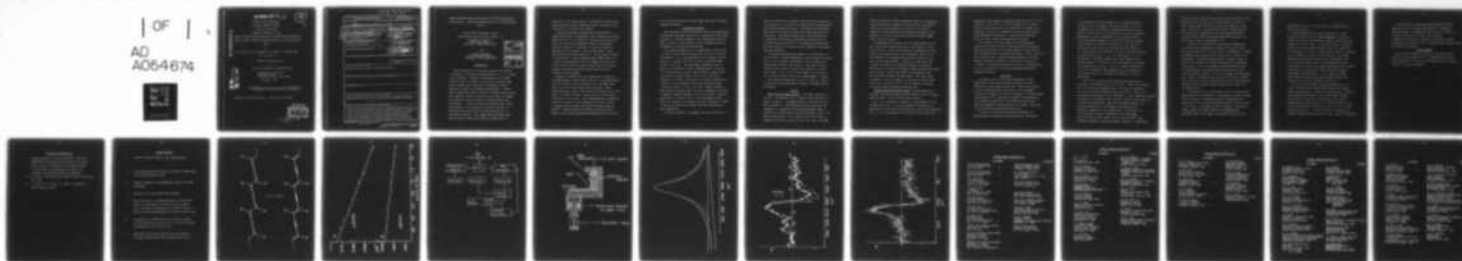APR 78   C T TZINIS, R H BAUGHMAN, S K BAHL   N00014-75-C-0883
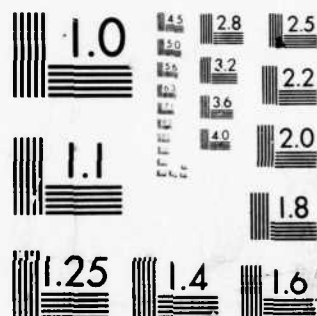UNCLASSIFIED   TR-78-01

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Sample Modulated Raman Spectroscopy and Frequency Modulated

Visible Light: Resonance Raman Spectrum of a Polydiacetylene

Fiber

by

C. T. Tzinis, R. H. Baughman, S. K. Bahl, P. Davidson and

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Certain Raman-active vibrational modes of polydiacetylene fibers shift in frequency as they are stretched. Thus, the frequency of the Raman-scattered visible light itself shifts as the fiber sample is subjected to a tensile stress. In this work a fiber has been subjected to a periodic tensile stress, and the frequency of the Raman scattered light was found to be modulated at the frequency of this stress. When the Raman signal was detected phase sensitively and frequency-locked at the stress modulation

405-436

Sample Modulated Raman Spectroscopy and Frequency Modulated

Visible Light: Resonance Raman Spectrum of a Polydiacetylene

Fiber

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Introduction

Certain Raman-active vibrational modes of any material
are sensitive to external stresses applied to it. The change
in the frequency, intensity or polarization of the Raman
scattered light as a result of the stress is usually small.
Often a rather great stress must be applied to cause a
significant change in the Raman band parameters. However,
where a significant spectral change in a Raman band does occur
upon applying a stress, in the low strain region the change
can be made to occur periodically by applying the stress
periodically. If this is done and the Raman signal, which
is modulated at the frequency of the applied stress, is
detected phase sensitively, the resulting spectrum is a
differential spectrum. This sample modulated Raman (SMR)
spectrum should contain only bands for modes which are

sensitive to the stress applied. Furthermore, the root mean square amplitude of the differential band is related to the sensitivity of the mode to the stress.

Although modulation techniques have been applied in Raman spectroscopy, they generally involve modulation of some characteristic of the incident or scattered light while the sample is essentially unchanged[1] during the measurement. Similarly, modulation techniques, in which the sample is modulated, are well known in optical reflectance, transmission and related spectroscopies.[2] In this paper we report the observation of sample modulated Raman (SMR) spectra of a highly oriented polymer fiber and suggest the application of the method to other systems for which SMR could provide valuable information about molecular manifestations of externally applied stress.

Recently we showed[3] that the frequencies of several of the Raman bands of single crystal fibers of a polydiacetylene shift as a tensile stress is applied along the fiber axis, which is also the polymer-chain axis direction. These frequencies were found to vary linarly with stress in the 0-2% range. As shown in Figure 1, shifts of up to 30 cm^{-1} were found for the $\nu(C\equiv C)$ mode. This polydiacetylene, hereinafter referred to as HDU, has the composition $(R-C_4-R)_n$, where R is $C_6H_5-NH-OCOCH_2-$ and has the overall structure shown in Figure 2. The HDU fibers were chosen for this work both because of the large stress-induced spectral shift of certain

of its bands and because the laser Raman spectrum is strongly resonantly enhanced.

Experimental Method

The experiment involves stressing an HDU fiber periodically and detecting the Raman signal that is modulated at the stress frequency. A schematic diagram of the experimental setup is shown in Figure 3, and involves a fairly standard electronic arrangement for modulation spectroscopy.

The transducer makes use of a piezoelectric crystal (PZT) to convert the periodic electrical wave form into a periodic displacement (i.e., a displacement which is linearly related to the a.c. voltage) and thus to apply the periodic axial stress to the fiber. This PZT-based transducer, shown in Figure 4, is designed to apply a maximum stress on the order of 0.1% which should thereby, cause a shift of about 2 cm^{-1} in the $\nu(C{\equiv}C)$ band and about 1 cm^{-1} in the $\nu(C{=}C)$ band frequency. In order to achieve this, we used a piezoelectric crystal of 2.54 cm diameter and 0.16 cm thickness having a material constant of 3.75 x 10^{-8} cm/V. Using a peak-to-peak driving voltage $\underline{ca}$ 1000v across the thickness of the PZT, a change in diameter (length) of 0.01% is obtained. This was amplified by the factor 10 by a lever arrangement, which, while not necessary in principle, was more convenient in order to obtain the desired 0.1% stress than other approaches in these experiments.

As noted in Figure 3, the signal from the oscillator of

the lock-in amplifier provided the wave form which shaped the voltage applied to the PZT by the Burleigh RC-42 ramp generator and thereby stressed the sample. The sample was illuminated with ca 4mw of 632.8nm radiation from a Spectra-Physics 124 He-Ne laser. The Raman scattered light was collected into a Jarrell-Ash 25-300 Raman spectrometer and detected with an ITT-FW-130 photomultiplier. The signal was preamplified and then detected by a PAR-124A lock-in amplifier using phase sensitive detection.

The detected and amplified signal is ΔI (see Figure 6), the difference in Raman intensities in the stressed and relaxed cycles, and is measured continuously at each frequency the monochromator passes to the detector. The fiber was given a small initial stress to length $(\ell+\Delta\ell)$ which is the zero-point length. Thus, in the "stress phase" of the cycle it went to length $(\ell+2\Delta\ell)$ and in the "relaxed phase" to length ℓ. The approximate magnitude of $\Delta\ell$ is 4×10^{-4} cm. When ΔI is plotted versus monochromator frequency, the SMR spectrum is observed.

Results

Sample Modulated Raman Spectra. The Raman spectrum of a static stressed HDU fiber is shown in the 2070-2120 cm^{-1} region in Figure 5. The SMR spectrum in the same region is shown in Figure 6. The Raman band in this region has been assigned[3] to a vibration of the polymer backbone which is essentially a carbon-carbon triple bond stretch, $\nu(C\equiv C)$. The Raman and SMR spectra in the region of the $\nu(C=C)$ band of backgone are similar to those for the $\nu(C\equiv C)$ band. The SMR

spectra displayed in these figures were taken at a modulating
(sample stretching) frequency of 224 Hz with a time constant
of 30 sec and a spectrometer scan rate of 0.5 $cm^{-1}sec^{-1}$.
Essentially similar spectra were obtained with smaller time
constant (i.e. 10 sec), the only difference being a higher
noise level (see Figure 7).

No SMR bands were observed for other Raman bands for the
HDU fiber under the conditions used. This probably is due
to two factors. First, the vibrations corresponding to other
bands are not significantly stress-dependent, which is the
case expected for the modes associated with bonds that are
not in the polymer backbone. Second, the laser power used
is quite low (4mW) so the dominant features in the Raman
spectrum are those that are resonantly enhanced while the others
are very weak. Source laser powers of much greater than
5mW at 632.8nm decompose the HDU fibers. With other types
of samples it could be feasible to increase the source power
greatly (by a factor of 10^2 to 10^3, for example, resulting
in stronger Raman and therefore SMR spectra.

Frequency Modulated Visible Light. The SMR experiment is
successful because the Raman scattered light is itself frequency
modulated (FM). This FM visible light occurs at
$\nu = \nu_o - \nu_i + (\delta\nu_i)\sin(2\pi f\, t)$ where ν_o is the frequency of the
incident laser line, ν_i is the vibrational frequency of the
bond when the fiber is at its zero point in the "stress-relaxation"
cycle, $(\delta\nu_i)$ is the magnitude of the shift in the vibrational

frequency at the maximum or minimum in that cycle, and f is the frequency at which the a.c. stress is applied to the fiber. Since the phase sensitive lock-in amplifier is locked to the frequency f and the d.c. component is rejected, only visible light modulated at frequency f is amplified and detected. Thus, FM visible light is obtained by this method.

The intensity of the FM light has not been measured quantitatively with all fiber, source and optical parameters varied and controlled, but is high enough that these spectra were obtainable with an incident laser power of 4 mW at 632.8 nm. The scattered light is highly polarized because of the high degree of molecular orientation in the fiber. The ability to obtain a differential spectrum with such low incident power is due to the fact that the Raman bands being studied are resonantly enhanced.

Discussion

An interpretation of the stress dependence of these Raman bands of HDU fibers has been presented previously, and need not be discussed further here. What is of interest is that the SMR spectrum is the derivative of the Raman spectrum with respect to stress on the sample. The quantitative interpretation of the SMR spectrum requires considering the way in which the magnitude of the signal from the lock-in amplifier depends on the properties of the sample (fiber) and of the optical and electronic devices used.

In order to illustrate which features dominate the SMR spectrum, we consider the problem from the interaction

of the radiation with this sample to the amplification and
detection of the FM light by the lock-in system. To do so,
we assume first the simplest time-dependent external pertur-
bation, a square wave, to the system, and study the resulting
SMR spectrum. And, we assume that the relaxation time in
the two processes (expansion of the PZT crystal and therefore
of the fiber in the positive half period of the "driving"
signal and contraction of the fiber and the PZT crystal in
the negative half) are negligible, and the intensities detected
by the PM tube are $I_{R-}(\nu_o - \nu_i - \delta\nu)$ and $I_{R+}(\nu_o - \nu_i + \delta\nu)$,
respectively. The FM scattered light, after amplification
and phase-sensitive detection, is the difference (ΔI_R) between
the intensities I_{R-} and I_{R+}. Now, it is easily shown that if
$\delta\nu$ is small compared to the bandwidth, ΔI_R is proportional to
the first derivative of the Raman intensity with respect to
Raman shift.

The effect of each of these simplifying assumptions on
the shape of the SMR spectrum is as follows. First, the relaxation
time is not entirely negligible and is different in magnitude
for the two processes (expansion and contraction). The use of a
sine-wave driving signal, of low frequency f, in the experiment reduces
the effects of relaxation times different from zero, but it does not
eliminate them completely. When the PZT element expands, the
relaxation properties of the system are dominated by the
relaxation properties of the ceramic PZT. In the other half
of the period the crystal contracts and the fiber itself
drives the whole system (lever, etc.) in such direction that
the fiber attempts to reach its original length (ℓ). Undoubtedly

the fiber would contract very fast (ca. with the velocity of the
sound in perfect crystals) if it did not have to rotate the
lever system, which has a mass at least 10^4 greater than the
fiber. The result of these effects is that the SMR spectrum
is distorted and unsymmetrical, and the maximum SMR signal
is recorded with phase difference between driving signal and
FM scattered light of approximately 13°.

Secondly, the effect of the fluorescence background is
to shift the whole SMR spectrum relative to the zero signal
level and broaden one of the two lobes of the SMR band. Although
the SMR spectrum crosses the zero close to the frequency that
corresponds to the maximum Raman signal, it doesn't cross at
exactly that frequency, as would be expected if no background
were present. Third, the asymmetry of the Raman band itself
affects the shape of the SMR spectrum by giving rise to two
unequal bands (the sharper the curve, the bigger the SMR signal).
And, finally, the imperfection of the fibers causes a broaden-
ing to the lower frequency (more shifted) side of the Raman band,
and that also contributes toward increasing the asymmetry of
the SMR spectrum.

The signal level is in the range $1-5\mu V$, so the resulting
SMR spectrum is not noise-free. For systems with larger induced
spectral shifts, or systems which can be illuminated with
higher power, the differential signal will have a better signal
to noise ratio. The spectrum given in Figure 7 is calculated
using a square wave signal and all the approximations
mentioned above. Even though it does not coincide with the

experimental curve, it is very close to it and explains its qualitative features.

The SMR spectra should contain bands only for those Raman-active modes which are stress-dependent, and thereby should "pick out" the bonds whose vibrations show them to be responsive to this stress. In principle, then, it should be possible to find out which molecular features are important in responding to a stress in a very complex material, something that is difficult to study by other methods. The obvious examples are biological systems, such as membranes, which have different characteristics (and presumably molecular structures) in stressed and unstressed conditions, have complex compositions and vibrational spectra, and which require measurement in aqueous media. It is feasible that SMR spectroscopy could show which bonds change, and how they change when the system responds to the stress.

The application of SMR to polymer problems and to the study of absorbed and adsorbed species should be straight-forward and productive. However, the application to materials whose morphology and moduli are such that great stresses would have to be applied to large samples will present technological problems that may not be worth solving just to obtain a single SMR spectrum when a series of static measure-ments would suffice. For many systems of biological and chemical interest SMR spectra may provide a useful new approach.

This method for obtaining frequency modulated visible
light may prove to have many applications in information
transfer systems. Although the intensity of this light is
not high, the ability to obtain it at selected
frequencies is attractive. Moreover, the intensity should
be improvable by several orders of magnitude with other materials
and higher source powers.

Acknowledgments

Footnotes and References

1. A spinning sample (in other experiments), where the
    sampleholder's configuration and action has the same
    effect as would a mechanical or polarization chopper
    in the incident or exciting beam, is taken to be
    unchanged (not modulated) during the experiment.

2. M. Cardona, "Modulation Spectroscopy" Academic Press, New
    York, 1969.

3. V. K. Mitra, W. M. Risen, Jr., and R. H. Baughman, J.
    Chem. Phys. <u>66</u>, 6 (1977).

Figure Captions

1.   Schematic bonding diagram of the polydiacetylene.

2.   Stress dependences of the 2105- and 1498 cm^{-1} Raman bands
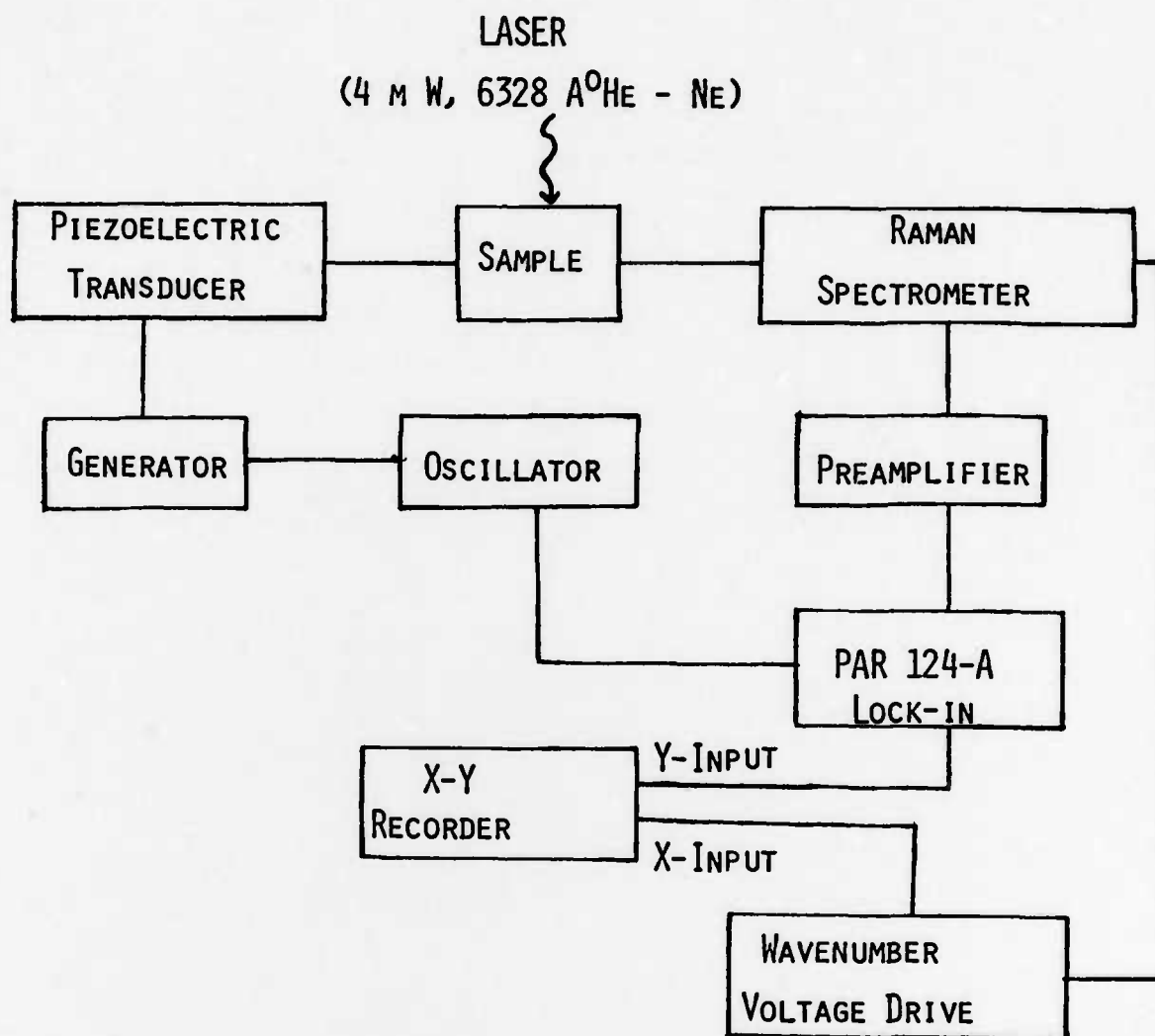   of HDU polydiacetylene fibers.

3.   Schematic diagram of the experimental setup for the SMR
     experiments.

4.   Diagram of the stress modulation transducer.

5.   *Raman spectrum of a stressed HDU fiber in the 2100 cm^{-1}*
     region, (a) experimental curve (solid line), and (b)
     curve calculated by Lorentzian fit to obtain function
     used in calculating SMR spectrum of Figure 6 (dotted line)

6.   SMR spectrum of the HDU fiber in the 2100 cm^{-1} region,
     (a) experimental curve (solid line), (b) curve calculated
     as described in text (dotted line).

7.   SMR spectra of HDU fiber taken with time constants of
     10 seconds (dotted line) and 30 seconds (solid line).

a.

ν (C≡C)

b.

ν (C=C)

2110

2100

2090

2080

cm⁻¹

1500

1490

1480

$\Delta L/L \times 100$

0.2 0.4 0.6 0.8 1.0 1.2 1.4 1.6

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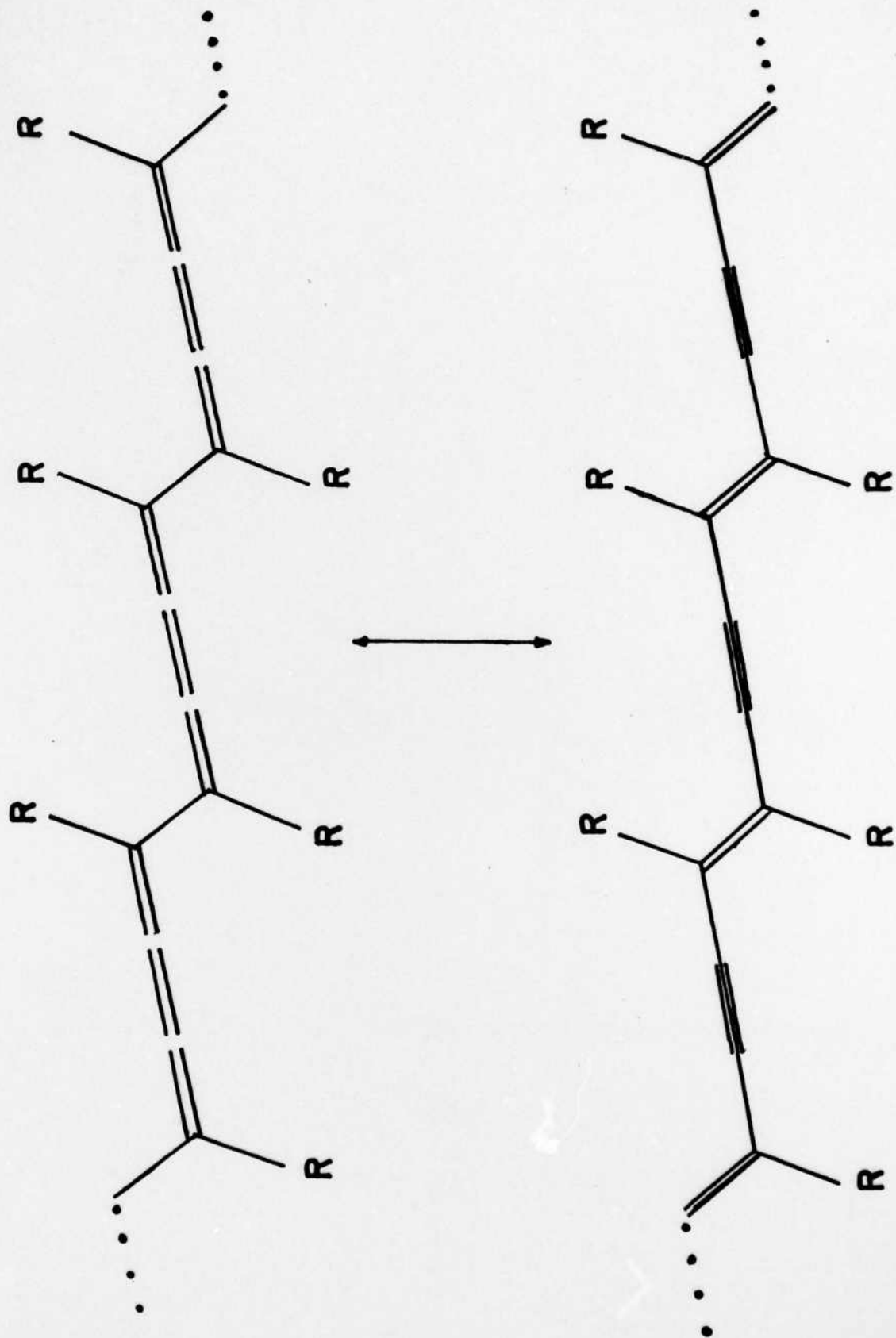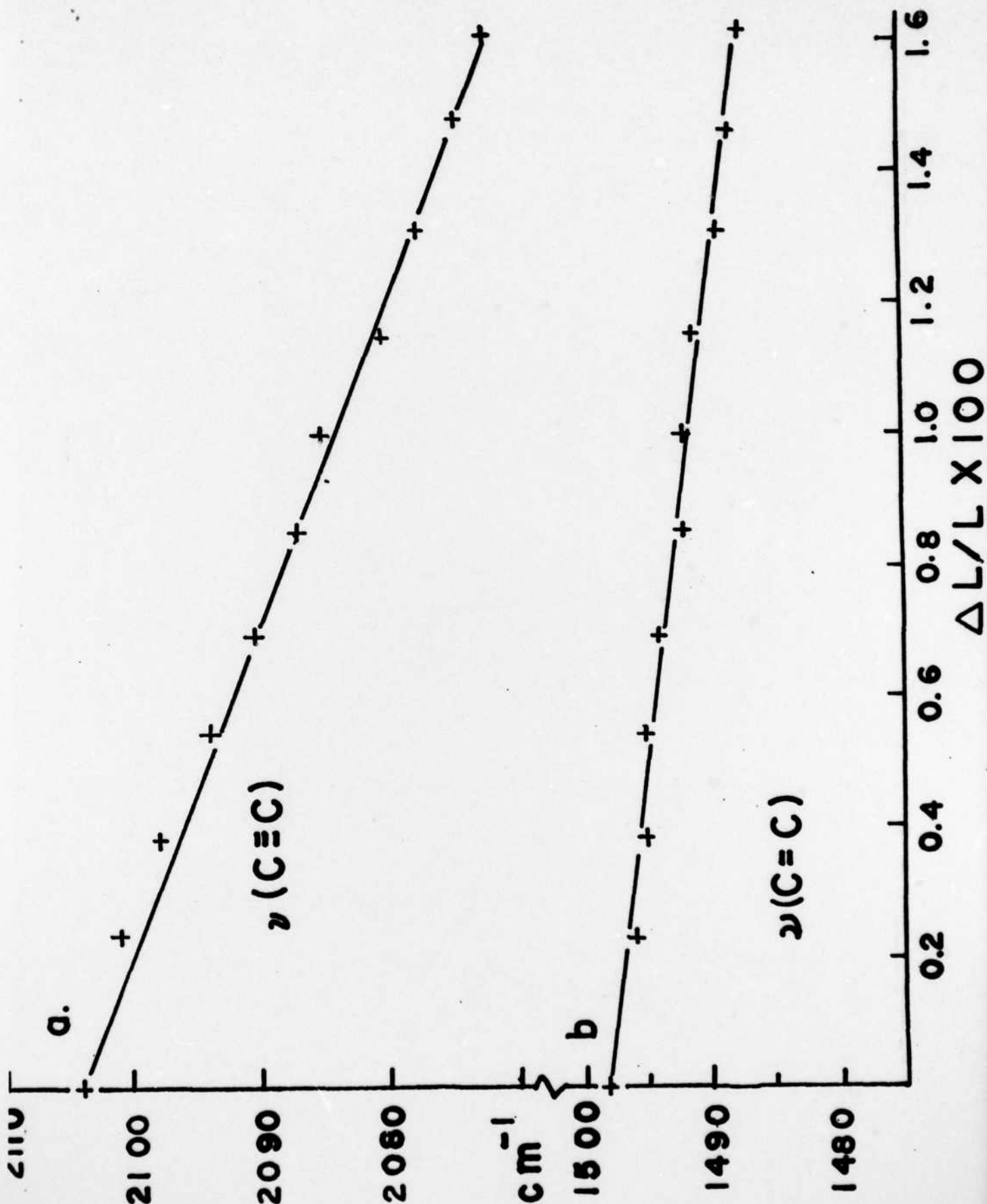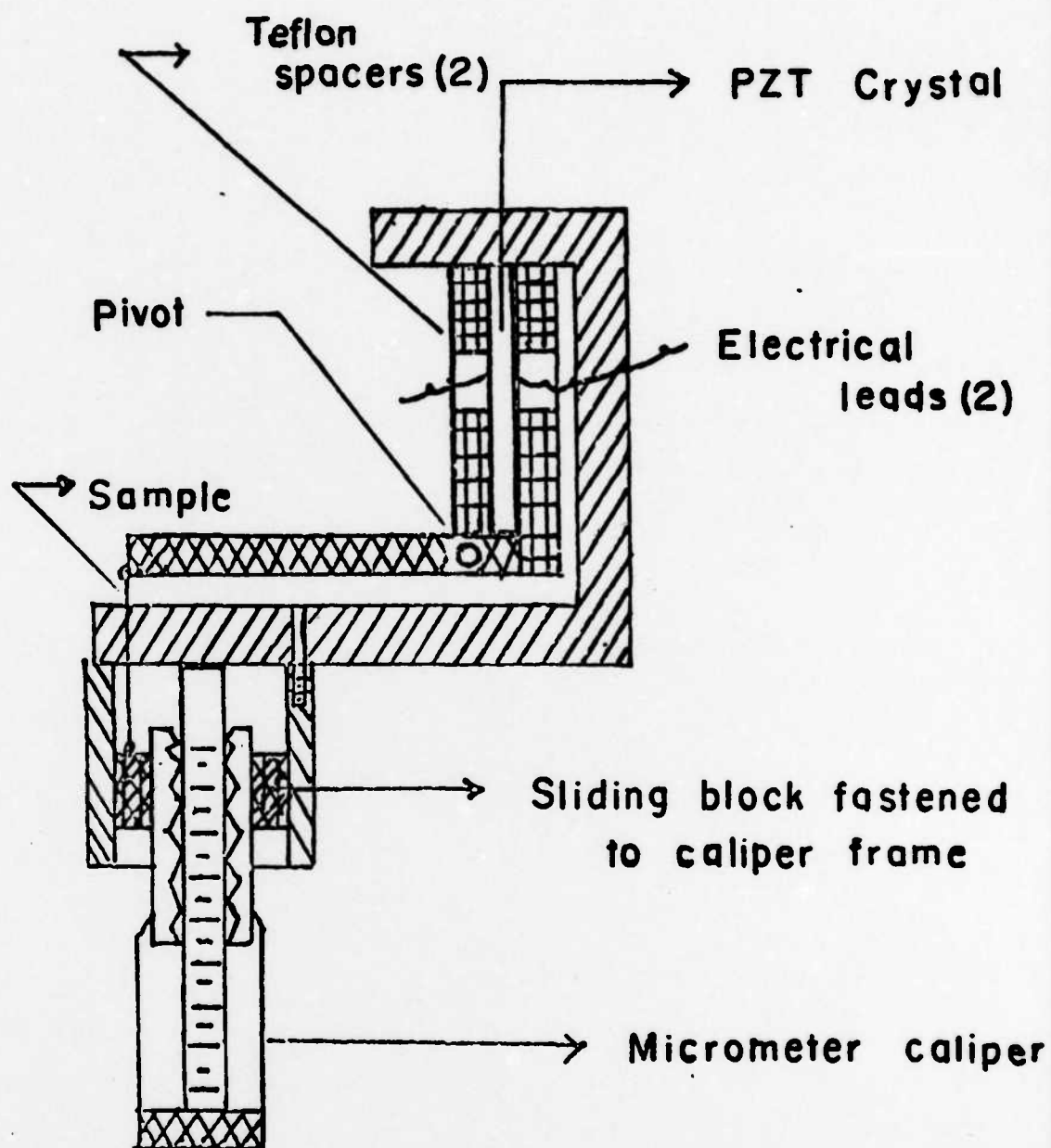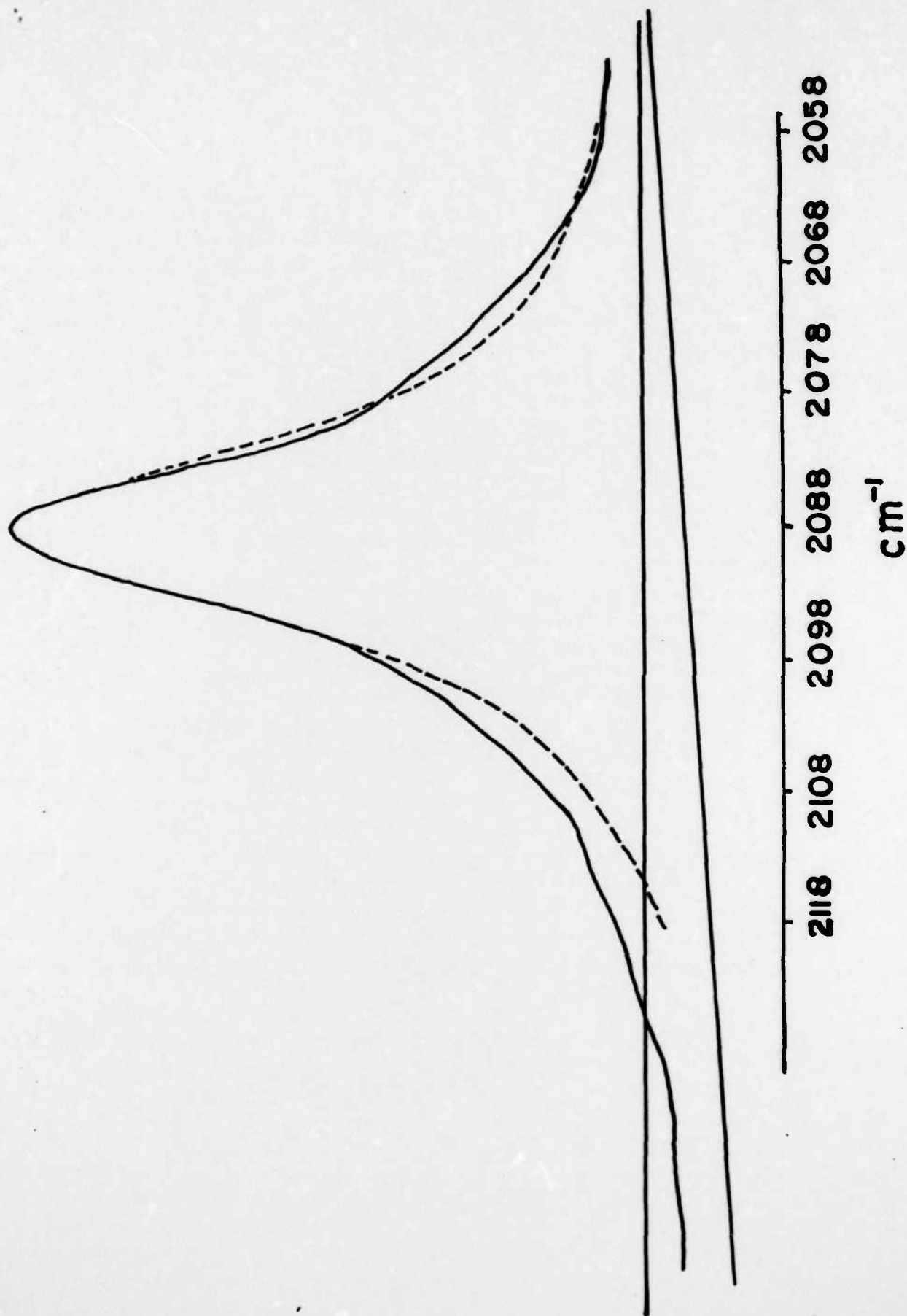
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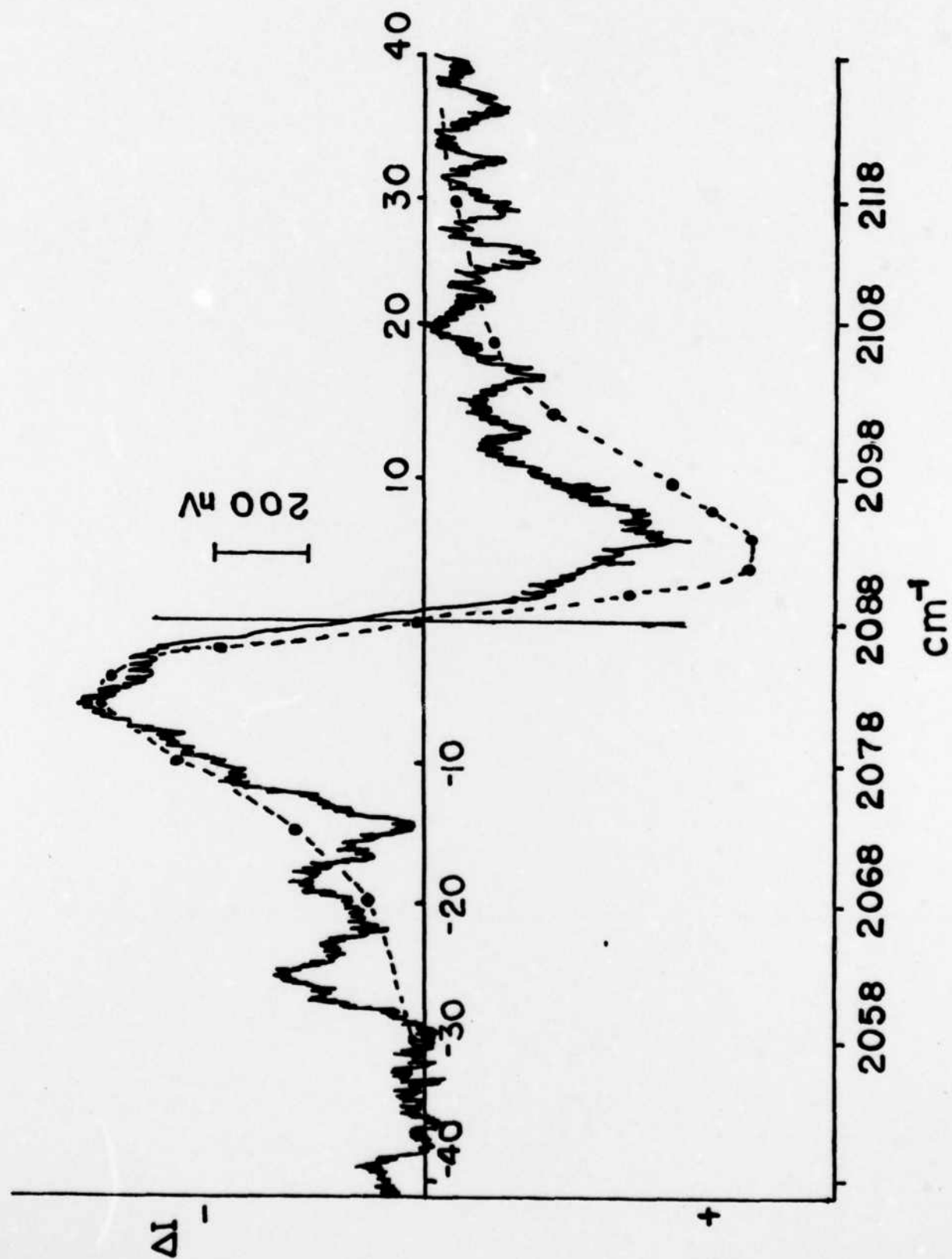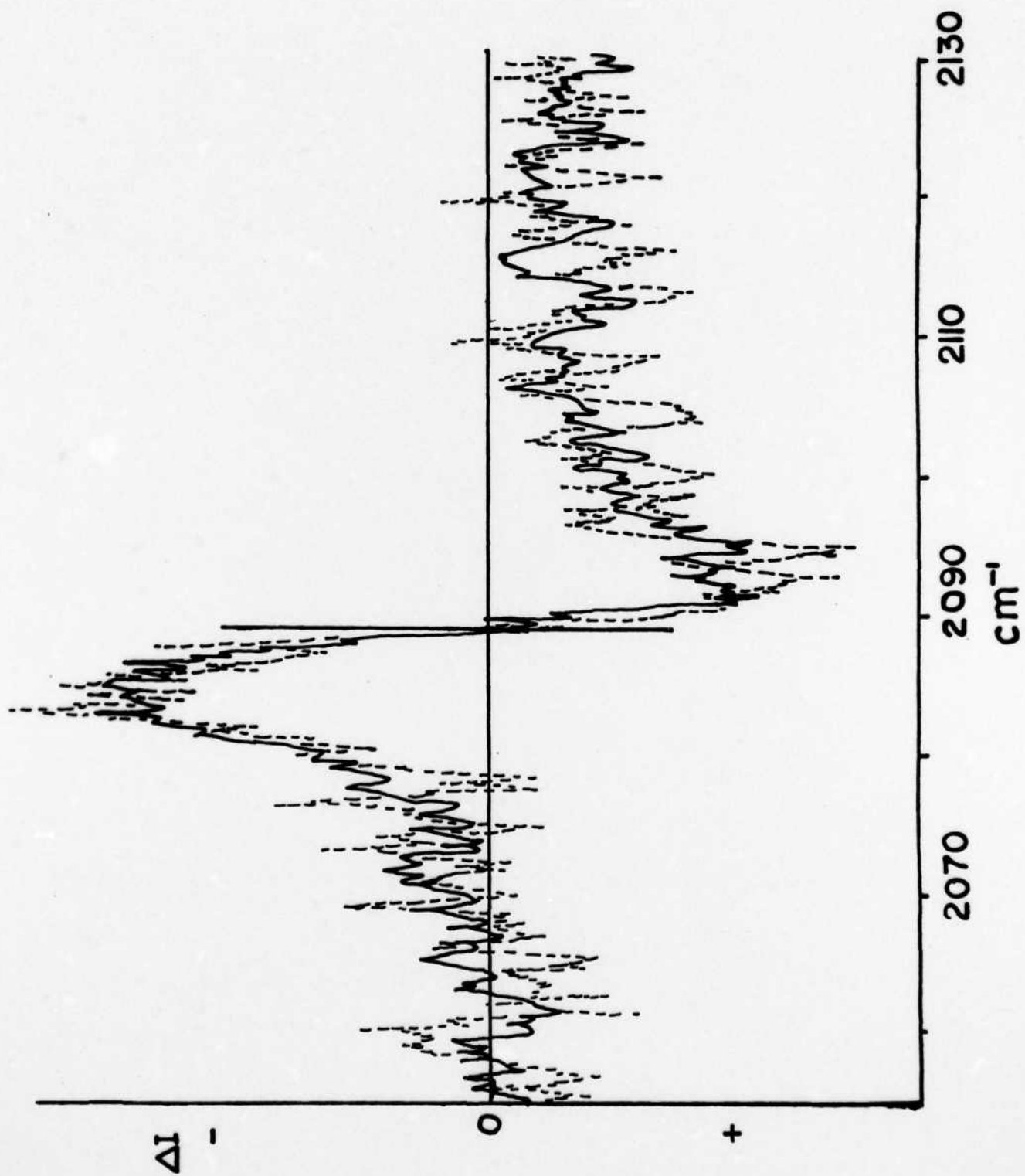Arlington, Virginia 22217
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78